

REMARKS

Applicant's attorney is appreciative of the interview granted by the Examiner on September 22, 2003. At that interview, the terminology to which objection has been raised was discussed, and it was agreed that a new substitute specification would be submitted.

Objection has been raised to the abstract on conflicting grounds, first that the application does not contain an abstract (page 2 of Office Action) and second, that the application contains two abstracts (page 5 of office action).

An abstract was added to the application in the paper filed on August 13, 2002, and the originally filed abstract was canceled in the paper filed on March 12, 2003. Thus a single abstract is in the application, and Applicant submits that this abstract is clearly written.

Regarding the drawings, new Figures 1 and 2 have been submitted. In these figures:

Fig. 1 has been labeled "Prior Art;"
"M2" in Fig. 1 has been removed;
"10" in Fig. 2 has been removed; and
"V" in Fig. 2 has been removed.

Moreover, in Example 1 of the specification, "100" now designates "underflow," and in Example 3, "An" has been changed to "In."

Based on the above changes, Applicant believes that all objections to the drawings have been removed.

Objection has been raised to the amendment of August 13, 2002 as containing new matter under 35 USC 132. The substitute specification filed on August 13, 2002 has been withdrawn, and a new substitute specification is filed herewith. In the new specification, the objected to terminology has been withdrawn, except for the designation of

solid content as "g/ l aluminate." As explained at the interview, this designation is identical to the original French text, and describes the solid content in the slurry. The original translation as g aluminate/ liter was an incorrect translation.

In addition, it is noted the designation "CPFT" for the amount of solid smaller than a given size has been removed in the new substitute specification.

The other changes in the substitute specification are minor in nature, and the new substitute specification does not contain new matter.

Claims 7-14 have been rejected under 35 USC 112, 1st paragraph, as containing subject matter not adequately described in the specification.

The claims have now been extensively amended to remove terminology to which objection has been raised, and to clarify the process steps. In this regard, "CPFT" has been removed from the claims, and has been replaced by "percent [or particles] finer than..." in accordance with the explanation given in the specification on page 3. It is not known whether there are still objections to claim 7, but it is noted that the process of determining the relationship R is discussed in detail in the originally filed specification at page 8, lines 11-31, and the discussion of trigger thresholds is found on page 9, lines 9-23 of the originally filed specification.

The limitations of claims 8, 9, 11, 13 and 14 have been objected to as being new matter. In this regard, it is noted that claim 8 corresponds to claim 2 as filed and claim 9 corresponds to claim 3 as filed. Claim 11 is supported by Example 1 at the top of page 14 of the originally filed specification.

Claim 13 is a recitation of steps a1)-a4) on pages 7-8 of

the specification, and claim 14 is a recitation of steps b1) - b4) on page 10 of the specification.

All claims have been amended to the extent possible to use terminology found in the specification as originally filed.

Withdrawal of this rejection is requested.

A further rejection of claims 7-14 under 35 USC 112, 1st paragraph, has also been made.

A question has been raised as to how the relationship R is determined in claim 7, with the comment that it does not appear to be based on CPFT X1 and CPFT X2, in view of the specification, page 7, that it is based on %<X2(t) and %<X1(t-t). However, these disclosures are actually the same, since CPFT X1 is the same as %<X1, and CPFT X2 is the same as %<X2. Claim 7 has been amended to remove "CPFT" and recites instead that the "percent smaller than" is measured over time (t) to determine R. Thus, there is no conflict.

Moreover, the recitations regarding "controlling the circuit" and "updating the correlation" (now "forming a correlation) are explained in detail in the explanation of Example 2 on page 16 of the specification.

Withdrawal of this rejection is requested.

Claims 7-14 have been rejected under 35 USC 112, 2nd paragraph, as indefinite.

In claim 7, "predetermined" has been alleged to be indefinite based on the *Seagram* case from 1950. As discussed at the interview, Applicants do not believe that this case is an accurate reflection of the current state of patent law and practice, and should not be cited.

In claim 7, the terms "regularly" and "updating" have been deleted.

As to the "corrective action," claim 7 has been amended

to clarify that this step brings the amount finer than X2 within the maximum permissible variation previously defined in the claim. This process is explained in the specification on page 6.

The corrective action itself may be adjustment of the solid content of the slurry, or other actions explained on page 6, lines 16-24.

Claim 9 has been amended to recite modifying amounts of liquor added to the first feed tank and first agglomeration tank, as explained at page 6, lines 25-29. Thus, the amount fed to each tank may be varied to achieve a desired result.

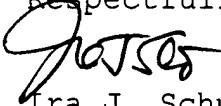
In claims 13 and 14, the "feed tank series" has been changed to "circuit" to provide antecedent basis.

The other objection to claim 13 is not understood, since, as explained above, there is no conflict between "CPFT" and "%<X."

As to the concept of "accidental phenomenon," this is explained in the specification on page 8, lines 2-4. Thus, an event may be observed on both of the determined curves, separated by a period of time, and this separation is used to determine a relationship between the two curves.

Withdrawal of this rejection is requested.

In view of the foregoing amendments and remarks, Applicant submits that the present application is now in condition for allowance. An early allowance of the application with amended claims is earnestly solicited.

Respectfully submitted,

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PROCESS FOR CONTROLLING THE PARTICLE SIZE IN A BAYER
CIRCUIT PRECIPITATION SYSTEM, INCLUDING AN
AGGLOMERATION PHASE

Technical domain Field of the invention

The invention relates to the precipitation of alumina trihydrate according to the Bayer process, carried out in an American type precipitation system including a preliminary agglomeration phase.

5

State of prior art Description of related art

The Bayer process can produce alumina from bauxite ore, particularly alumina designed to be transformed
10 into aluminum by igneous electrolysis. According to this process, the bauxite ore is treated when hot by means of an aqueous sodium hydroxide solution with an appropriate concentration in order to obtain a suspension containing pregnant sodium aluminate liquor
15 and insoluble residues. After separation of these residues, the pregnant sodium aluminate liquor, also called Bayer liquor, is decomposed by seeding with recycled aluminum trihydroxide particles until aluminum trihydroxide grains (also called alumina trihydrate or hydrargillite) are obtained; and which are themselves
20 then calcinated to obtain an alumina with particular particle size distribution and physicochemical properties. The sodium aluminate liquor depleted in alumina (spent liquor) is then recycled to digest the
25 ore, possibly after being concentrated, by the evaporation and addition of sodium hydroxide or caustic soda.

The productivity of the liquor during its crystallisation is defined by the quantity of alumina restored in the form of alumina trihydrate by crystallisation of the pregnant liquor, related to a given volume of pregnant liquor. The productivity expressed in kilograms of alumina per cubic meter of liquor ($\text{kg of Al}_2\text{O}_3/\text{m}^3$), is related to the caustic concentration in the pregnant liquor. In general, this concentration in American type Bayer processes is close to 100-130 g of $\text{Na}_2\text{O}/\text{liter}$, which is lower than in European type Bayer processes, and this explains why a productivity in the crystallisation of the pregnant liquor is considered to be good when it exceeds 70 kg of $\text{Al}_2\text{O}_3/\text{m}^3$ for an American type Bayer process, or when it exceeds 80 kg of $\text{Al}_2\text{O}_3/\text{m}^3$ for a European type Bayer process.

The difference between European type and American type Bayer processes is in the solid content of the slurry during precipitation. The slurry is the result of introducing a recycled alumina trihydrate seed into the aluminate liquor, and part of the alumina in solution changing to the solid phase. We will define the solid content in the slurry as the weight of solid particles present in the slurry per unit volume of pregnant aluminate liquor entering into the precipitation workshop (and not per unit volume of the suspension).

Alumina to be transformed into aluminum by igneous electrolysis must have a number of properties, including:

- o good pourability flowability so that electrolysis tanks can be continuously supplied with controlled quantities of alumina,

- a high dissolution rate,
- a low tendency to dusting.

These properties are closely related to the morphology and the particle size distribution of alumina grains, themselves closely related to the morphology and particle size distribution of hydrargillite particles formed during the precipitation. It is particularly important to limit the proportion of very fine particles that can be classified in two main categories: fines (for which the average diameter is between 10 and 50 μm) and ultrafines, for which the average diameter is less than 10 μm . Since a good correlation is observed between the particle size distribution of alumina and the particle size distribution of the production hydrate from which it is derived, an attempt is made to control the particle size of the ~~rotating~~ hydrate circulating used in the precipitation series, and particularly in the crystal growth phase. Thus for example, in order to obtain a good quality "metallurgical" alumina, an attempt is made to obtain a ~~rotating~~ circulating hydrate for which the amount passing a 45 μm sieve is less than 10%, ~~in other words i.e.~~ a suspension containing less than 10% of particles with a diameter of less than 45 μm . In the rest of this discussion, we will denote the quantity passing X micrometers as "% $<\!X$ ".

Concerning the American type Bayer process, the precipitation comprises a preliminary agglomeration phase characterized by a particularly low solid content. In patent US 4 234 559, the precipitation circuit comprises firstly a series of agglomeration tanks and then a series of feed tanks and finally three

classification tanks (primary, secondary, tertiary). While the hydrate produced is derived from the underflow from the primary classification tank, the fines seed originating from the underflow from the 5 tertiary classification tank is inserted in controlled quantities into the series of agglomeration tanks and the larger seed originating from the underflow from the secondary classification tank is added into the series of feed tanks. Since the fines are destroyed during 10 the agglomeration phase, the problem of controlling the particle size of the hydrate produced does not arise.

But attempts are always still being made to increase the productivity of the American type Bayer process, which is lower than the productivity of 15 European type Bayer cycles. In US 5 158 577 and EP 0 515 407, only part of the pregnant liquor is added into the agglomeration system tank series, and the rest is added directly to the crystal growth. This can give 20 result in different solid contents in these two parts of the crystallization system, a low solids content of dry material in agglomeration tanks which is essential if it is required that agglomeration should take place under good conditions, and a high solid content in feed tanks which can increase productivity.

25 But an instability in the precipitation is observed if the solid content of dry material is increased at the feed and if the number of agglomeration tanks is limited, with a serious risk of the sudden appearance of large quantities of fines in the rotating 30 circulating hydrate (particle size crisis). This type of crisis should be avoided, since if it is not corrected no correction is made, the particle size

quality of the produced hydrate is strongly deteriorated.

The particle size instability is due to a reduction in the ratio between the sum of the production hydrate and the fine seed sent to agglomeration, and the rotating circulating hydrate. A reduction in this ratio makes it impossible to implement effective corrective actions when a drift of the amount passing 45 µm (% < 45) is observed on the rotating circulating hydrate.

Statement of the problem Summary of the invention

Therefore, the applicant attempted to define a process that could increase the productivity of the American type Bayer process by preventing unacceptable particle size fluctuations, particularly the sudden appearance of large quantities of fines and ultrafines in the rotating circulating hydrate.

20

Purpose of the invention

The process developed by the applicant is a process for controlling the precipitation of an American type Bayer circuit including a preliminary agglomeration phase, a crystal growth phase and a classification phase, in which the particle size quality of the hydrate produced is monitored by measuring the amount of rotating hydrate passing X2 µm in feed tanks, characterized in that it comprises:

- a) a preparation step carried out once and for all, intended firstly to setup a relation R in intensity and in time between rotating

- 5 circulating hydrate material passing X1 μm and material passing X2 μm , where X1 is less than X2, and secondly to define trigger thresholds on the value of material passing X1 μm , starting from the maximum authorized variation interval on values passing X2 μm ;
- 10 b) control of the process itself, carried out during the installation operating period which, apart from the daily measurement of material passing X2 μm and a regular update of the correlation between the said material passing X2 μm and the particle size of the hydrate produced, a daily measurement of the ~~rotating~~ circulating hydrate passing X1 μm and a regular update of the relation R between the said material passing X1 μm and the said material passing X2 μm , and triggering of corrective action in the slurry at the beginning of the precipitation when the measured value of material passing X1 μm reaches one of the regularly updated trigger thresholds determined in the previous step.
- 15 20 25 30

This corrective action in the slurry at the beginning of precipitation may be a modification to the temperature of the aluminate liquor added into the agglomeration tank, the addition of additives at the beginning of the crystal growth system such as "Crystal Growth Modifiers" described in US 4 737 352, recycling a part of the end of ~~crystallisation~~ crystallization slurry, or preferably, modification of the solid content in the slurry in the first agglomeration tank.

The solid content in the slurry in the agglomeration phase may be modified simply by adding

more or less pregnant aluminate liquor in the first agglomeration tank, the remaining aliquot being directed to the feed tank. If the quantity of material passing $X_1 \mu\text{m}$ is too great, there are too many fines; 5 the amount of pregnant aluminate liquor fed into the agglomeration tank must be increased. If the amount passing $X_1 \mu\text{m}$ is too low, there is a risk that productivity will drop; the pregnant aluminate liquor feed from the agglomeration tank ~~needs to~~ must be reduced. 10

The preliminary step, carried out once only, is intended to determine the relation R and trigger thresholds for the values of $X_1 \mu\text{m}$ that will be used at the beginning of application of the process control.

15 This preliminary step comprises the following steps:

- a1) Daily measurement of material passing $X_1 \mu\text{m}$ in the slurry at a particular point in the precipitation system, which is used to produce a first particle size time diagram represented by a curve $Y = \% < X_1(t)$. 20
- a2) Daily measurement of material passing $X_2 \mu\text{m}$ in the slurry at a particular point in the precipitation system, which is used to produce a second particle size time diagram represented by a curve $Y = \% < X_2(t)$ and in which X_2 , greater than X_1 , is a value already known for its good correlation with the particle size of the hydrate produced. For example, it may be material passing $45 \mu\text{m}$ measured in slurry at pump-off. 25
- a3) Creation of an empirical relation between the two particle size time diagrams, the purpose of which is to characterize the relation between 30

the variation of the population of material passing $X_1 \mu\text{m}$ and the variation of the population of material passing $X_2 \mu\text{m}$, in intensity and in time. This relation R may be written in the form:

$$R(\%<X_2(t), \%<X_1(t-\tau)) = 0$$

where t is the date on which material passing $X_2 \mu\text{m}$ is measured and τ is a characteristic time interval estimated by observing the occurrence of the same accidental phenomenon on each curve (the same type of extreme, an inflection, etc.).

a4) Definition of the maximum threshold and the minimum threshold of material passing $X_1 \mu\text{m}$, obtained from the relation R previously established and a maximum interval of the authorized variation of values of material passing $X_2 \mu\text{m}$.

The purpose of the relation R is to predict the variation in the particle size, in other words to anticipate crises, by observing the variation in the population of the finest hydrate particles (with which have a size of less than $X_1 \mu\text{m}$). The applicant observed that material passing $X_1 \mu\text{m}$ anticipates enables anticipating a change in the material passing a higher X_2 value; an accident on the time diagram $\%<X_1(t)$ is amplified and shifted in time on the $\%<X_2(t)$ time diagram. The time shift " τ " is higher as the difference between X_1 and X_2 increases. In practice, a value X_2 greater than 40 μm (normally 45 μm) will be chosen, and the value of X_1 will be taken to be less than or equal to 20 μm .

The measurement point is preferably at pump-off, but it may take place earlier, provided that it remains within the crystal growth series. Measurement points for material passing X1 and for material passing X2 may 5 be different. However, they must remain the same throughout the process control and must be as far as possible from the points at which disturbing additions are made irregularly in the slurry.

Measurements referred to as being "daily" are 10 regular measurements and although they are not necessarily daily, they are sufficient frequent to give useable time diagrams.

Concerning the required variation interval of material passing X2 μm , a maximum is defined above 15 which it is known that the particle size of the hydrate produced is no longer satisfactory (too many fines) and a minimum is defined below which it is known that economic operating conditions become bad.

The trigger thresholds are thus determined from the 20 time diagram for the material passing X1 μm , taking account not only of the maximum authorized variation interval on the values of material passing X2 μm , but also the uncertainty of the measurement of material passing X1 μm and the stability of the efficiency of 25 the hydrate classification system.

This preparatory step, at the end of which the time diagrams, the relation R and the trigger threshold become operational, is of the order of three months. But this step can be accelerated either by deliberately 30 triggering an excess creation of fines, or by analyzing previous particle size results if they contain the required information about material passing X1 and X2 μm .

Since precipitation is a complex phenomenon that depends on a large number of parameters (particularly the composition of the treated bauxite that may change in time), instead of using a relation R established 5 once and for all, it is better to use a relation that is regularly updated. Similarly, the correlation between the value of material passing $X_2 \mu\text{m}$ and the particle size of the hydrate produced must be updated regularly. This regular updating may be applied at a 10 less frequent rate than daily measurements (for example monthly).

The actual process control comprises the following phases:

- 15 b1) A daily measurement of the material passing $X_1 \mu\text{m}$ in the slurry at a particular point in the precipitation system, in order to complete the first particle size time diagram represented by the curve $Y = \% < X_1(t)$.
- 20 b2) A daily measurement of the material passing $X_2 \mu\text{m}$ in the slurry at a particular point in the precipitation system, in order to complete the first particle size time diagram represented by the curve $Y = \% < X_2(t)$.
- 25 b3) Regular updating (for example monthly) of the empirical relation R between the two particle size time diagrams and the definition of trigger thresholds of material passing $X_1 \mu\text{m}$, or updating after an important modification in a process parameter.
- 30 b4) Triggering of a corrective action in the slurry at the beginning of the precipitation when the measured value of material passing $X_1 \mu\text{m}$ reaches one of the thresholds defined in b3).

This corrective action is preferably a modification to the solid content in the slurry in the first agglomeration tank. If ~~it is a~~ the maximum threshold is reached, there are ~~two~~ too many fines, the ~~and the~~ 5 ~~solid content of dry material~~ is reduced by introducing a stronger aliquot of the pregnant aluminate liquor into the first agglomeration tank. If ~~it is a~~ the minimum threshold is reached, the feed of pregnant aluminate liquor into the agglomeration tank is reduced 10 and the ~~crystal growth feed~~ into feed tanks is increased.

As for the preparatory step, empirical relations are determined starting from daily particle size measurements made on the ~~rotating circulating~~ hydrate. 15 As for the preparatory step, the daily rate is not necessarily daily, but is sufficiently frequent to be able to produce useable time diagrams. Trigger thresholds on the "X1 μm passing material" curve are also deduced from the particle size time diagram; they 20 take account of the uncertainty of the measurement of material passing X1, the maximum authorized variation interval on values of material passing X2 μm , and the stability of the efficiency of the hydrate classification system.

25 For relatively low solid content ($\approx 350 \text{ g/l}$ ~~aluminate of aluminate/liter~~), it is sufficient to measure amount passing $X1 = 20 \mu\text{m}$. This may be done using instruments that diffract laser beams and make mass determinations (Malvern Mastersizer, Cilas, etc.).

30 But in order to increase productivity, much higher solid content must be achieved, comparable to those achieved in European type processes, in other words greater than 700 g/l aluminate of aluminate/liter.

However, as the solid content in feed tanks increases, the need for a low value of X_1 also increases. When the solid content is high, a longer time is necessary to correct particle size disturbances; therefore, it
5 is necessary to have a greater time shift τ ; and this time shift is proportional to the difference between X_1 and X_2 .

In this case, it is preferable to measure the amount passing 10 μm , or even a lower value (down to
10 1.5 μm). When X_1 becomes this low, it becomes necessary to use a celloscopic measurement (COULTER or ELZONE) that determines counts rather than mass. These measurements are more difficult, but the economic consequences are negligible.

15 Productivity can also be increased by increasing the caustic content of the aluminate liquor. The applicant has observed that this method of controlling the particle size can be applied very well on industrial installations with concentrations reaching
20 160 g of Na_2O / liter.

Concerning the corrective action, it is also recommended that an empirical relation should be set up in advance to quantify the effects of the said action, for example during the preparatory phase. Thus, in
25 order to quantify the effects of the modification to the solid content in the first agglomeration tank, the first step will be to establish the actual relation between the said solid content and the proportion of destroyed fines.

30 **Brief description of the drawings**

Fig. 1 is a schematic diagram of a prior art Bayer circuit; and

Fig. 2 is a schematic diagram of a Bayer circuit according to the invention.

Embodiments of the invention - Examples

5 The embodiment of the invention will be better understood from the following description of a number of examples.

Figures 1 and 2 show the part of the Bayer circuit corresponding to the precipitation phase, the precipitation being of the American type with a preliminary agglomeration step.

10 Figure 1 illustrates the first example typical of prior art.

Figure 2 illustrates subsequent examples describing 15 two embodiments of this invention.

These embodiments are described in examples 2 and 3. To simplify the presentation, we have illustrated the classification phase will with all conventional classification devices in example 1. In fact, and 20 particularly with the increase in the solid content, an expert in the subject one of ordinary skill in the art could select additional or replacement systems such as cyclones or filters.

25 Example 1 (Prior art)

The pregnant aluminate liquor 1 enters the precipitation circuit at a temperature of about 75°C, enters the first agglomeration tank A. It has a caustic concentration of 130 g of Na₂O/ liter.

30 The tertiary seed 9, composed essentially of fine and ultrafine particles, is added into the first agglomeration tank A and is mixed with pregnant liquor. The resulting slurry 2 passes through a sequence of

agglomeration tanks such that practically all fines and ultrafines have disappeared after an average residence time of about 5 hours. During the agglomeration phase, the solid content in the slurry has increased from
5 20 g/l aluminate of aluminate / liter to about 30 g/l aluminate of aluminate / liter.

At the exit from the agglomeration series, the slurry **3** is added into the first feed tank **N** with the secondary seed **8**, and the addition increases the solid
10 content by 80 g/l aluminate of aluminate / liter. The new slurry **4** passes through a sequence of feed tanks with an average residence time of about 20 hours, and cooling by about 5°C. At the exit from the feed, the slurry **5**, called the "pump-off", ~~contains~~ has a solid
15 content of about 175 g/l aluminate of dry aluminate material / liter. By measuring the amount of this slurry **5** passing 45 µm at the exit from the crystal growth **M1**, the particle size quality of the production hydrate can be estimated so that an optimum variation
20 interval can be defined for this material passing 45 µm. Under the conditions in the example, approximately 10% of material passing 45 µm will give a good quality "metallurgical" alumina.

The slurry **5** at the exit from the crystal growth is
25 then added into the first classifier tank **PT**. The overflow underflow **100** from the first classifier **PT** produces production hydrate and the overflow **6** that has a solid content of 100 g/ l aluminate of aluminate / liter, is added into a second classifier tank **ST**. The
30 underflow from the second classifier tank **ST** acts as a secondary seed **8** that is reinjected at the beginning of the crystal growth **N** and the overflow **7**, which then has

a solid content of only 20 g/liter, is sent to a third classifier tank TT. The overflow 10 from the third classifier tank is the spent aluminum liquor that is reinjected as the bauxite green liquor at the beginning 5 of the Bayer cycle, after concentration by evaporation and the addition of caustic soda. The overflow 9 from the third classifier tank is added back as the tertiary seed into the first agglomeration tank A after filtration and washing.

10

Example 2: Precipitation according to the invention with an average solid content in the rotating circulating hydrate

15 The pregnant aluminate liquor 1 that enters the precipitation circuit at a temperature of about 75°C, is separated into two aliquots, the first aliquot 1a representing about one third of the total liquor being added into the first agglomeration tank A, and the 20 second aliquot 1n being added into the first feed tank N. The caustic concentration in the pregnant aluminate liquor 1 is 130 g of Na₂O/Na₂O/liter.

The tertiary seed 9a, composed essentially of fine and ultrafine particles, is added into a first 25 agglomeration tank A after filtration and partial or complete washing, and is mixed with the pregnant liquor. The resulting suspension 2 follows a sequence of agglomerating tanks such that a predefined proportion of fines and ultrafines has disappeared 30 after an average residence time of 5 hours. During the agglomeration phase, the quantity of hydrate is

increased by 10 g/l aluminate of aluminate / liter entering the workshop (1a and 1n).

The slurry 3 at the exit from the agglomeration series is added into the first feed tank N with the 5 second aliquot 1n of pregnant aluminate liquor, the rest of the washed or unwashed tertiary seed 9n, washed or unwashed, and the secondary seed 8, the addition of which increases the solid content by 220 g/l aluminate of aluminate / liter. The new slurry 4 follows a 10 series of feed tanks with an average residence time of 20 hours and cooling of 5°C. At the exit from the crystal growth, the slurry 5 contains has a solid content of about 350 g/l aluminate of dry aluminate material / liter.

15 The slurry 5 at the exit from the crystal growth is added into a first classifier tank PT. The underflow 100 from the first classifier PT supplies the production hydrate, and the overflow 6 which has a solid content of 250 g/l aluminate of aluminate / liter 20 is added into a second classifier tank ST. The underflow from the second classifier tank ST acts as a secondary seed 8 that is reinjected at the beginning of the crystal growth N and the overflow 7, which then only has a solid content of 30 g/ liter, is sent to the 25 third classifier tank TT.

A measurement M1 of the material passing 45 µm, and a measurement M2 of the material passing 20 µm, are made in the slurry every day at pump-off, using a laser diffraction apparatus.

30 Observations of the particle size quality of the hydrate produced can be used to define the authorized variation interval on values of material passing 45 µm,

and the target set value C on the material passing 20 µm is defined, by means of the empirical relation defined during the preparation phase and continually updated afterwards.

5. Depending on the difference between M₂ and the value of the set value C defined in advance to guarantee the particle size quality of the product and the differences M₂ - C obtained in the previous days, the quantity of fines to be agglomerated in addition to
10 or less than the previous day, are determined.

During the preparation phase, the relation between the solid content in the agglomeration phase and the proportion of destroyed fines was determined. This relation is used to fix the aliquot 1a used in the
15 agglomeration. This aliquot 1a fixes the solid content in the agglomeration tanks and therefore the change required for destruction of the fines.

Furthermore, starting from measurements M₁ and M₂ made on previous days, the relation between the set
20 value C and the required level M₁ for the material passing 45 µm, and secondly the relation between the solid content and fines destroyed in the agglomeration, are adjusted.

- 25 **Example 3: Precipitation according to the invention with a high solid content in the rotating circulating hydrate**

The pregnant aluminate liquor 1 arriving in the precipitation circuit at a temperature of about 75°C,
30 is separated in two aliquots, the first 1a representing about half of the total liquor being added into the first agglomeration tank A, the second 1n being added

into the first feed tank N. The caustic concentration in the pregnant liquor **1a** is 130 g of Na_2O / liter.

The filtered tertiary seed **9**, composed essentially of fine and ultrafine particles, is added into the 5 agglomeration step **A** and mixed with the fraction **1a** of pregnant liquor. The resulting slurry **2** stays in the agglomeration phase for about 5 hours. The proportion between the quantity of tertiary seed and the flow of liquor **1a** is adjusted such that a predefined proportion 10 of fines and ultrafines disappears. The quantity of hydrate during the agglomeration phase is increased by 15 g/l ~~of aluminate entering in the workshop / liter~~ (**1a** and **An 1n**).

At the exit from the agglomeration series, the 15 slurry **3** is added into the first feed tank N and with the second aliquot **1n** of pregnant aluminate liquor and the secondary seed **8**, the addition of which increases the solid content by 840 g/l ~~aluminate of aluminate / liter~~. The new slurry **4** passes through a series of 20 feed tanks with an average residence time of 18 hours and cooling by 10°C. At the exit from the crystal growth, the slurry **5** ~~contains has a solid content of~~ about 1000 g/l ~~aluminate of solid aluminate content / liter~~.

25 At least one measurement **M1** of the material passing 45 µm is made every day at this location using a laser diffraction apparatus, and a measurement **M2** of the material passing 1.5 µm is made with an ELZONE counter.

The slurry **5** at the exit from the crystal growth is 30 added into a first classifier tank **PT**. The underflow **100** from the first classifier **PT** supplies the production hydrate and the overflow **6**, that has a solid

aluminate content of 870 g/l aluminate liter, is added into a second classifier tank **ST**. The underflow from the second classifier tank **ST** is used as a secondary seed **8** that is reinjected at the beginning of the 5 crystal growth **N** after filtration and the overflow **7**, that has a solid content that no longer exceeds 30 g/liter, is sent to the third classifier tank **TT**.

The process for monitoring and controlling the particle size of the ~~rotating~~ hydrate in the slurry at 10 pump-off is identical to that described in example 2.

Advantages of the process according to the invention

The process according to the invention has the following advantages:

- Particle size fluctuations related to the increase in
5 the inertia of the system originating from the increase in solid contents are avoided.
- The productivity of liquors can thus be increased:
 - by controlling crystallization at high solid contents without endangering the quality of the
10 alumina produced,
 - by adjusting the pump-off particle size to the maximum level compatible with quality requirements and the production classification system.